

### REMARKS

The Notification of Defective Response stated that a translation of the application must be submitted, because the current translation of the Application is defective. Specifically, the Notice states that the Claims must be filed as translated from original Application, before changes are made by a preliminary amendment.

In response, Applicant encloses herewith a substitute translation which overcomes the issue raised by the Notification.

Furthermore, Applicant hereby resubmits the Amendment originally submitted on May 13, 2004, reflecting the changes in the substitute translation and requests withdrawal of the Amendment submitted on December 5, 2005.

Favorable consideration on the merits is requested.

Respectfully submitted  
for Applicant,

By:



D.W. Darren Kang  
Registration No. 51,859  
McGLEW AND TUTTLE, P.C.

JJM/DWK:

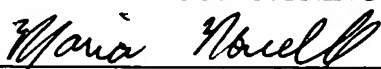
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SHOULD ANY OTHER FEE BE REQUIRED, THE PATENT AND TRADEMARK OFFICE IS HEREBY REQUESTED TO CHARGE SUCH FEE TO OUR DEPOSIT ACCOUNT 13-0410.

I HEREBY CERTIFY THAT THIS CORRESPONDENCE IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE AS EXPRESS MAIL IN AN ENVELOPE ADDRESSED TO: COMMISSIONER FOR PATENTS, P.O. BOX 1450, ALEXANDRIA, VA 22313-1450, NO. EV435294116US

Maria Nowell

NAME OF PERSON SIGNING CERTIFICATION



SIGNATURE

DATED: January 27, 2006  
McGLEW AND TUTTLE, P.C.  
BOX 9227 SCARBOROUGH STATION  
SCARBOROUGH, NEW YORK 10510-9227  
TELEPHONE: (914) 941-5600



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**Method of Producing Alloy Ingots**

Description

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The invention relates to a novel fusion-metallurgical method of producing, at a low cost, ingots of metallic and intermetallic alloys of high chemical and structural homogeneity, in particular ingots of  $\gamma$ -TiAl.

- 10 In aeronautics and astronautics and in motor racing,  $\gamma$ -TiAl-based intermetallic alloys have proceeded from a laboratory development stage to industrial application since 2000. Advantageously combined high-temperature and low-weight properties ensure their use in aeronautics and astronautics. High-temperature and corrosion resistance recommend the material for use
- 15 in rapidly movable components of engines, for example valves in combustion engines or blades in gas turbines. The properties of this material depend on chemical and structural homogeneity to an extent not known so far in structural materials. Consequently, manufacturing ingots of correspondingly high quality is technically highly complicated and costly. Homogeneous ingots are needed in various process routes for the manufacture of
- 20 further semi-finished products or components of TiAl as starting material (see H. Clemens and H. Kestler (2000), Advanced Engineering Materials 9, 551; Y.-W. Kim (1994), JOM 46 (7), 30; and P.A. Bartolotta and D.L. Krause (1999) in Gamma Titanium Aluminides, ed. Y.-W. Kim, D.M. Dimiduk and M.H. Loretto, (TMS Warrendale, PA, USA 1999), 3-10).
- 25

The  $\gamma$ -TiAl-based technical alloys presently used are of multiphase structure, containing the ordered hexagonal  $\alpha_2$ Ti<sub>3</sub>Al, typically at a proportion of

5 to 15 volume percent, in addition to ordered tetragonal  $\gamma$ -TiAl as a main phase. Refractory metals as alloying elements can lead to the formation of a metastable body-centered cubic (bcc) phase which appears either as a  $\beta$  phase (disordered) or as a B2-phase (ordered). These alloying additions  
5 improve oxidation resistance and creep strength. Inferior quantities of Si, B and C serve for increased strength of the cast structure (see B. Inkson and H. Clemens (1999), MRS Symp. Proc. 552, KK3.12; S. Huang, E. Hall, D. Shuh (1991), ISIJ Internatioal 31 (10), 1100 and Y.-W. Kim and D.M. Dimiduk (1991), JOM 8, 40). Corresponding C contents can lead to precipita-  
10 tion hardening (see V. Güther, A. Otto, H. Kestler and H. Clemens, (1999) in Gamma Titanium Aluminides, ed. Y.-W. Kim, D.M. Dimiduk and M.H. Loretto, (TMS Warrendale, PA, USA 1999), 225-230). The alloying elements Cr, Mn and V increase room temperature ductility of the otherwise very brittle TiAl. Depending on the field of application, alloy development  
15 has led to a number of alloy variants which will be described in detail below.

TiAl alloys are customarily produced as ingots by multiple remelting in a vacuum-arc furnace (see Fig. 1) (VAR – vacuum arc remelting). A pressed  
20 electrode, which includes all alloying constituents, is melted off, expanding in diameter. Fundamental problems result from occurring inhomogeneities in the alloy composition of the  $\gamma$ -TiAl ingot. A comparison of the Al contents in twice or triple remelted  $\gamma$ -TiAl ingot material reveals that local fluctuation of the Al contents in the range of  $\pm 2$  atomic percent are still  
25 observed in twice remelted  $\gamma$ -TiAl ingot (see Fig. 2). Triple remelting in the VAR mill is necessary for obtaining sufficient alloy homogeneity (see V. Güther, A. Otto, H. Kestler and H. Clemens, (1999) in Gamma Titanium Aluminides, edl Y.-W. Kim, D.M. Dimiduk and M.H. Lorettok, (TMS

Warrendale, PA, USA 1999), 225-230; V. Güther, Properties, processing and applications of  $\gamma$ -TiAl, Proc. 9<sup>th</sup> Ti World Conference, 08-11.06.1999, St. Petersburg and V. Güther, H. Kestler, H. Clemens and R. Gerling, Recent Improvements in  $\gamma$ -TiAl Ingot Metallurgy, Proc. Of the Aeromat 2000  
5 Conference and Exhibition, (Seattle, WA, June 2000).

Contrary to titanium alloys (ingot diameter up to 1.5 m), workable diameters in the case of  $\gamma$ -TiAl are distinctly restricted by reason of limited formability. Presently, there is a predominant demand on the market for ingots  
10 of only approximately 200 mm of diameter.

By using VAR technology, there is an increase in diameter of approximately 40 mm per remelting job. Aiming at a final diameter of approximately 200 mm, this means that the process must proceed from pressed  
15 electrodes of about 60 mm of diameter maximally, the porosity of which is in the range of 40 percent. The small diameter limits the strength of the pressed electrode, and thus the possibly employable length, to approximately 1.5 m (which corresponds to a total mass of approximately 18 kg). The smaller the diameters of the first pressed electrode, the higher the cost  
20 of manufacture, because less material can be melted per melting cycle. A triple remelted VAR ingot of a diameter of 180 mm and a length of 1000 mm – corresponding to prior art industrial embodiment -will need an overall of 10 melting jobs (6 initial melting jobs, 3 second melting jobs, 1 third melting job), which causes some costs. The loss of material (piping  
25 etc.) per ingot is presently 35 percent. Moreover, the conventional manufacturing method does not offer any flexibility in the choice of ingot diameter.

Alternatives of manufacturing titanium alloy ingots are cold hearth electron-beam melting and plasma arc cold hearth melting (PACHM). While electron-beam melting (see Fig. 3 top) has been used solely for pure unalloyed titanium, the PACHM method (see Fig. 3 bottom) is used for the  
5 production of titanium alloys and also of  $\gamma$ -TiAl ingots. In this case the starting material is melted in a cold crucible by a plasma torch and the liquid melt is supplied via a plasma-torch heated channel system to an equally plasma heated billet discharge. This method has led to insufficient alloy homogeneity, which may be due to the limits of the method (see W. Porter,  
10 Proceedings of 3<sup>rd</sup> Int. Symp. Structural Intermetallics, ed. K.J. Hemker et al., TMS Warrendale 2001, page 201). Even the addition of an induction coil for improved homogenization of the melt in the plasma heated billet discharge did not show the desired results (see M. Loretto, Titanium 95, Science and Technologies; A.L. Dowson et al., in Gamma Titanium Aluminides (1995), ed. Y.-W. Kim, R. Wagner and M. Yamaguchi (TMS Warrendale, PA, USA 1995), 467-474; M. Volas, Industrial Initiatives in Wrought Orthorhombic and Gamma TiAl Mill Products; Proc. Of the Aeromat 2000 Conference and Exhibition, Seattle, WA, June 2000).  
15  
20 Furthermore, the production of  $\gamma$ -TiAl-based alloys by means of ingot casting from a cold wall induction or plasma furnace, or by means of inert gas spraying from a cold wall crucible, to  $\gamma$ -TiAl powder and powder metallurgical processing has been put into practice technologically. These alternatives have so far resulted in insufficient microstructure (porosity upon ingot  
25 casting) and excessive costs (powder metallurgy).

Reference is made to U.S. patents 5 846 351, 5 823 243, 5 746 846 and 5 492 574, standing in for the state of the art in VAR technology.

It is an object of the present invention to embody a method of reproducibly producing  $\gamma$ -TiAl ingots of high chemical homogeneity and little porosity, which can be put into practice more easily and at a lower cost than the VAR method specified above which needs numerous steps of melting for obtaining the desired high homogeneity and inferior porosity. Moreover, the method is intended to offer the possibility of arbitrarily dimensioning the alloy ingots within a range of what is technically reasonable by elusion of the VAR-method restrictions specified above.

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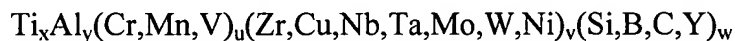
This object is attained in a method of producing metallic and intermetallic alloy ingots by continuous and quasi-continuous billet discharge from a cold wall induction crucible with the alloy material, in a molten or pre-homogenized state, being continuously or quasi-continuously supplied to a cold wall induction crucible (see Fig. 4). The method of continuous casting for the production of metallic and intermetallic alloy ingots of high homogeneity and inferior porosity is characterized by the following chronological steps:

- 20 (i) producing electrodes by customarily mixing and compressing the selected starting materials;
- (ii) at least once remelting the electrodes obtained in step (i) by a conventional fusion-metallurgical process;
- (iii) inductively melting off the electrodes obtained in steps (i) and (ii) in a high frequency coil;
- 25 (iv) homogenizing the melt obtained in step (iii) in a cold wall induction crucible; and

- (v) withdrawing the melt by cooling from the cold wall induction crucible of step (iv) in the form of solidified ingots of freely adjustable dimensions.
- 5 By alternative, the following sequence in the continuous casting method for the production of metallic and intermetallic alloy ingots of high homogeneity and inferior porosity can be put into practice (see Fig. 5):
- (i) producing electrodes by conventionally mixing and compressing the selected starting materials;
  - 10 (ii) at least once remelting the electrodes obtained in step (i) by a conventional fusion-metallurgical method;
  - (iii) producing a pre-homogenized, molten material from the electrode material obtained in step (ii) by melt-off in a cold crucible plasma furnace;
  - 15 (iv) homogenizing the melt obtained in step (iii) in a cold wall induction crucible; and
  - (v) withdrawing the melt, solidified by cooling, from the cold wall induction crucible of step (iv) in the form of cylindrical ingots of freely adjustable diameters and lengths.

20

The method is preferably used for the production of intermetallic  $\gamma$ -TiAl based alloy ingots, the alloys being generally specified by the following summation formula:



25

The concentrations of the alloying constituents are customarily within the following ranges (in atomic percent):

$$x = 100 - y - u - v - w$$

$y = 40$  to  $48$ , preferably  $44$  to  $48$

$u = 0.5$  to  $5$

$v = 0.1$  to  $10$  and

$w = 0.05$  to  $1$ .

5

Inductive melting of the electrodes in step (iii) takes place in a high frequency field of a frequency of preferably  $70$  to  $300$  kHz, in particular  $70$  to  $200$  kHz, and preferably at temperatures of  $1400^{\circ}\text{C}$  to  $1700^{\circ}\text{C}$ , in particular  $1400^{\circ}\text{C}$  to  $1600^{\circ}\text{C}$ . For uniform dropping to be obtained, the electrode is  
10 rotated, preferably at a speed of  $4$  rpm. The withdrawal speed of the electrode is continuously variable from  $0$  to  $200$  mm/min.

In the case of inductive melting, the method is preferably performed quasi-continuously, by one or several electrodes being fed quasi-continuously  
15 while an ingot is simultaneously withdrawn from the cold wall induction crucible.

Homogenization of the melt in the cold wall induction crucible of step (iv) takes place preferably by overheating at  $10$  to  $100$  K, preferably  $40$  to  
20  $60$  K. This corresponds to temperatures of  $1400^{\circ}\text{C}$  to  $1750^{\circ}\text{C}$ , preferably  $1450^{\circ}\text{C}$  to  $1700^{\circ}\text{C}$ , depending on alloy composition. The frequency range of the coil is  $4$  to  $20$  kHz, preferably  $4$  to  $12$  kHz.

Cooling the melt upon withdrawal of the ingots in step (v) preferably takes  
25 place by the aid of water-cooled copper segments, the diameters of the ingots preferably being in a range of  $40$  to  $350$  mm, by special preference  $140$  to  $220$  mm.



The withdrawal rates are adjustable between 5 to 10 mm/min. The withdrawal rate must be adapted to the dropping rate (step iii) which can be in the range of 50 kg/h.

- 5    The present method according to the invention enables novel intermetallic  $\gamma$ -TiAl-based alloy ingots to be produced which excel by a novel combination of dimensions on the one hand and homogeneity on the other. Therefore, the invention also relates to intermetallic  $\gamma$ -TiAl-based alloy ingots which are characterized by
- 10    (a) a length to diameter ratio of  $> 12$ ;  
      (b) homogeneity related to local fluctuations of the aluminum and titanium of  $< \pm 0.5$  atomic percent; further metallic alloying constituents:  $\pm 0.2$  atomic percent; non-metallic alloying additions (boron, carbon, silicon):  $\pm 0.05$  atomic percent.
- 15    The gist of the method according to the invention resides in the continuous or quasi-continuous supply of a pre-homogenized melt of the alloying material to a cold wall induction crucible (KIT). Within the scope of the present invention, it has surprisingly been found that melting off the electrode
- 20    material that serves for the production of metallic and intermetallic alloy ingots occasions considerable homogenization of the material so that a single subsequent step of homogenization in the cold wall induction crucible will do to obtain, by means of these two steps, as far as possible a degree of homogenization, which can be accomplished only by a comparatively great
- 25    number of remelting steps in the VAR method. Consequently, the method according to the invention is substantially less complicated and costly than the VAR method used so far.

The KIT loses its principal prior art function, namely melting material that is always supplied to the KIT in a solid state. An essential advantage of the method according to the invention resides in that segregation phenomena as a reason for inhomogeneities of the final material, which are always observed when solid alloys of multiphase structure are melted in the KIT, do not occur because the material arrives in a liquid state in the KIT.

Another advantage resides in that the frequency range of the induction coil, which is favorable for homogenization of the molten alloy, exceeds the frequency range that is favorable for melting a solid alloy. Surprisingly, this helps considerably reduce surface porosity of the ingot withdrawn from the solidifying melt in the KIT, improving ingot quality.

A special advantage of the method according to the invention resides in that any required dimensions of the alloy ingot can be put into practice by the dimensions of the cold wall induction crucible being freely selectable within a technologically reasonable scope, which is not ensured by VAR technology.

Vacuum or protective-gas execution of the method is preferred, and non-polluted production waste can be returned to the process. In accordance with the invention, material loss amounts to 12 percent as compared to 35 percent in conventional VAR technology.

The method according to the invention enables local (macroscopic) fluctuations of the main alloying elements, aluminum and titanium, of  $< \pm 0.5$  atomic percent to be put into practice throughout the ingot; further

metallic alloying constituents:  $\pm 0.2$  atomic percent; strength increasing elements (boron, carbon, silicon):  $\pm 0.05$  atomic percent.

The scope of the invention also comprises novel combinations of prior art  
5 sub-processes, known per se, which ensure a continuous or quasi continuous supply of liquid, pre-homogenized material to a cold wall induction crucible with the aim of continuous or quasi continuous billet withdrawal from the KIT.

10 This relates in particular to the combination of an inductively heated melt-off device for alloy rods and alloy electrodes (inductive drop melting), a KIT with a billet withdrawal equipment, and the combination of a plasma cold wall furnace with a heated channel system, of an overflow in the form of a skull, comprising said KIT and said billet withdrawal equipment. Both  
15 combinations of methods according to the invention will be described in detail below, taken in conjunction with exemplary embodiments.

Important steps of these combinations of methods according to the invention, such as inductive melting of electrodes, the PACHM method, the fusion of alloys in a cold wall induction crucible, and billet withdrawal of  
20 alloys from ceramic as well as cold wall induction crucibles, have been known and employed, accompanied with distinctly varying boundary conditions, aims and materials.

25 The inductive fusion of metals has been described for example in U.S. patents 4 923 508, 5 003 551 and 5 014 769. Moreover, inductively melting off electrodes has also been described in connection with the manufacture of titanium alloyed powder by the so-called EIGA (electrode induction

melting gas atomization) method (cf. DE-A-41 02 101, DE-A-196 31 582).  
In this method, an alloy electrode dips into an HF coil which is insulated by  
ceramics against arc-over. As in the present case, the electrode is com-  
pletely melted by a surface melting process. Further treatment of the melt  
5 takes place in a gas jet where the drops are atomized. This method serves  
exclusively for the production of powder and not for the production of in-  
gots. In the present description, the melt is subjected to further homogeni-  
zation in a KIT prior to billet withdrawal (production of ingots).

10 As regards any prior art concerned with the fusion of materials in a cold  
wall induction crucible, reference is made to U.S. patents 5 892 790 and  
6 144 690. Neither patent deals with the production of ingots, which is  
completely different with patents DE-A-198 52 747 and DE-A-196 50 856.  
The decisive difference between DE-A-198 52 747 and DE-A-196 50 856  
15 and the present invention resides in the supply of material. While pre-  
homogenized, molten material is supplied to the KIT in the present case,  
solid material is fed to the KIT in the mentioned patent. This means that, in  
the present case, energy input into the KIT serves exclusively for further  
homogenization and keeping the material liquid whereas, in the patent  
20 specified, melting, homogenizing and solidifying occur at the same place –  
the KIT. This increases the probability of segregation.

Ingots withdrawal is also known from the state of the art, in particular with-  
drawal from the ceramic crucible. The prior art patents predominantly re-  
late to ingot withdrawal of non-ferrous metals (Cu, brass). The above-  
25 mentioned patents DE-A-198 52 747 and DE-A-196 50 856 however com-  
prise ingot withdrawal from the cold wall induction crucible, with the ma-  
terial being fed in solid form, and not as pre-homogenized, molten mate-  
rial, to the KIT from which the ingot is withdrawn. This can lead to differ-

ences in homogeneity – as described above – in the material that is withdrawn as an ingot.

Electrode production takes place preferably by pressing and/or sintering  
5 powdery or granulated alloying constituents (cf. DE-A-196 31 582 to –584, DE-A-198 52 747).

In the drawings,

10 Fig. 1 is an illustration of the VAR process for multiply remelted  $\gamma$ -TiAl ingots: (1) electrode feed, (2) furnace chamber, (3) air-cooled current supply, (4) cable collecting duct, (5) electrode guide, (6) water-jacket crucible, (7) part of the vacuum arrangement, (8) XY\_adaption, (9) pressure pick-up;

15 Fig. 2 is an illustration of deviations of Al contents in the longitudinal direction of the ingot after double (black symbols) and triple (gray symbols) VAR remelting;

20 Fig. 3 is a diagrammatic view of cold wall electron beam melting (top) and cold wall plasma melting (bottom);

Fig. 4 is an illustration of the method according to the invention (Example 1) for the fabrication of chemically homogeneous  $\gamma$ -TiAl ingots  
25 of variable dimensions: (1) rotating electrode, (2) inductive HF coil, (3) cold wall induction crucible, and (4) cooling arrangement and ingot withdrawal;

Fig. 5 is an illustration of the method according to the invention (Example 2) for the fabrication of chemically homogeneous  $\gamma$ -TiAl ingots of variable dimensions: (1) charging slope, (2) plasma torch, (3) cold hearth, (4) cold wall induction crucible (KIT), and (5) cooling arrangement, and (6) ingot withdrawal.

In conclusion, the method according to the invention deals with fusion-metallurgical technology for the production of chemically and structurally homogeneous alloy ingots, in particular of  $\gamma$ -TiAl ingots as ingot material for the molding route or remelter stocks for the casting route. The technology comprises a combination of:

- the production of pre-homogenized, molten material by the aid of inductive melting in an HF coil or of the PACHM method. In both cases, the starting material comprises the sum of all alloying constituents which are however only insufficiently homogeneously distributed;
- the supply of molten material to a cold wall induction crucible;
- the further homogenization of the liquid (melted) material in the cold wall induction crucible (KIT); and
- the preferably continuous withdrawal from the KIT.

The individual steps of the method will be described in detail in the following.

Producing the electrodes takes place first. By the aid of a conventional fusion-metallurgical method, for example by VAR technology, pressed electrodes, which include all alloying constituents (Ti sponge, Al granules, pre-alloying granules), are melted off by enlargement of diameter, forming rods of a diameter of for instance 150 mm. These are rods of low chemical

homogeneity and of a certain porosity. They serve as electrodes for the ensuing billet withdrawal.

The first technological step can be illustrated in two alternative ways – by  
5 inductive melting or the PACHM method. Bother methods aim at the production of pre-homogenized, molten material.

In the case of inductive melting, the electrode, which has been melted off by a customary method, is inductively melted by the aid of an HF coil (according to the EIGA method, see DE-A-41 02 101, DE-A-196 31 582) in a  
10 KIT. The coil/drop-material system and the shape of the coil interact closely. In accordance with minimum demands on melting rates and ingot diameters, the outer-oscillating-circuit frequency range amounts to 70 to 300 kHz. When high-frequency induction fields are employed, a pronounced skin effect must be expected to occur in the melting electrode. In  
15 combination with the comparatively low heat conductivity of titanium aluminides, this effect leads to local overheating in the boundary layer and, subsequently, to aluminum evaporation that cannot be sensed quantitatively. Since the pronounced current flow in the skin layer is an essential  
20 feature of high-frequency alternating current fields and can therefore not be avoided, the only possibility of reducing aluminum evaporation resides in reducing the dwell time of the material in the electro-magnetic field. Uniform pre-heating of the dropping electrode by means of inductive heating (average frequency of approximately 500 Hz to 4 kHz) to temperatures  
25 below the melting point of the alloy will reduce the energy and capacity that is required in the field for melting by the amount of energy already inputted. This either reduces the dwell time in the a.c. field of a single volume element and in total of the entire dropping electrode, as a result of which the melting performance increases, or lower overall capacities of the

HF coil will be needed. The requirements and consequences explained show that design and dimensioning of the outer oscillating circuit and the HF frequency make sense only in close interaction with the designed electrode pre-heating capacity. Electrode feed rates must be adjustable within a  
5 scope that will allow dropping rates corresponding to mass flow rates of at least 50 kg/h in the case of electrode diameters of 150 mm.

In the case of the PACHM method, the melting process is put into practice by plasma torches, which have two functions: melting the starting material  
10 and keeping constant ambient conditions during ingot discharge. Starting material in the form of mechanically comminuted pre-alloyed compacts is charged successively via a hydraulic platform into the melting chamber. Finally, the material is melted by the aid of the plasma torches in the water-cooled cold wall crucible of copper. The cold wall crucible (cold hearth)  
15 serves as an instrument for the elimination of undesired high-density (furnace bottom) and low-density inclusions (floating slag) of the melt and as a reservoir for the supply with molten material of the crucible/ingot-withdrawal system. The amperages of the plasma torches above the cold hearth range between 275 to 550 A, but may vary depending on the type  
20 and number of plasma torches used.

In the ensuing step, the melt is fed to the cold wall induction crucible. In the KIT, which is equipped with a movable bottom, the homogeneity of the melt in a greater molten volume that is largely kept constant is further im-  
25 proved by the agitating effect of the electromagnetic field. The dwell time of the melt in the crucible amounts to approximately 20 min to 45 min. Skull melting in the cold wall induction crucible (KIT) is a technology that has become industrially established for years. Electromagnetic induction in a water-cooled copper crucible produces a field that is used for heating and



melting the materials. Simultaneously, the occurring Lorentz forces partially squeeze the melt off the crucible walls, establishing in the melt a circulating flow that will consequently lead to excellent mixing of the melting phase. In the vicinity of the crucible bottom and in the bottom part of the crucible wall, a specific, solid skull will develop, conditioned by the form of the electromagnetic field. In combination with the free surface produced by the Lorentz forces, this skull prevents any direct contact of the melt with the crucible, eliminating any risk of contamination throughout the melting phase and ensuring mill safety.

10

In the case of inductive melting, continuous supply of the KIT with melt is ensured by the connected electrode depot which can take up several electrodes at a time, which are then successively melted off. In the case of the PACHM method re-charging of mechanically comminuted, pre-alloyed material takes place by way of a hydraulic platform.

15

The bottom skull, the thickness and habit of which depend directly on the form of the induction field, offers a point of departure for the possibility of semi production. If the bottom is lowered in the course of the process, the system reacts in such a way that a renewed state of equilibrium tends to form by another layer growing on the previous bottom skull. Continuously lowering the bottom will lead to a system of steadily adapting states of equilibrium and, consequently, to an almost continuously growing bottom layer. Since the base of the bottom skull is defined by the bottom of the crucible, the growing of further layers will result in a semi-finished product (ingot) originating. However, the steady output of mass from the KIT also requires a supply of further molten material.

20

25

Cooling the melt upon ingot withdrawal preferably takes place by the aid of water-cooled Cu segments.

5 Ingot withdrawal from the KIT produces a chemically homogeneous and largely nonporous ingot. In this method, the diameter of the KIT is freely selectable to a major extent, offering variable selection of ingot diameters. Withdrawal rates will preferably be in a range of 0 to 50 mm/min.

10 The products manufactured according to the invention can be used for various purposes. Primarily, semi-finished products are made from them in a first step of transformation (extrusion), which are then used for being further worked in the transformation route (forging, rolling). Ingots of high structural and chemical quality are needed for the production of  $\gamma$ -TiAl-based components via the transformation route. These components may for  
15 instance be valves and turbine blades which must comply with a demand for excellent quality and highest requirements.

Furthermore, the products according to the invention may also serve as remelter stocks for the manufacture of cast blanks by precision casting and  
20 centrifugal casting. Remelter stocks are needed as starting material for the precision and centrifugal casting route. Chemical and structural quality does not predominate, because the material is melted once again – as opposed to ingots. Therefore, step (ii) can be omitted in the method according to the invention and the pressed electrodes can directly be melted induc-  
25 tively or, respectively, pre-mixed compacts can be melted by the PACHM method. The precision casting route serves for the production of components of complicated design and complex requirements. The  $\gamma$ -TiAl-based turbo charger, which has been commercialized, is mentioned here by way

of example. Centrifugal casting is a method of manufacturing at a low cost mass-produced components (for example valves) of simple design and requirements. Producing remelter stocks by the method according to the invention results in products that are distinctly more homogeneous than corresponding prior art products and, owing to the ingot withdrawal, can be manufactured in any cylindrical dimensioning, whereas the method used so far depends on the dimensions of the available mold. The method according to the invention enables the diameter and length of the remelter stocks to be freely selected, which is a simple way of making direct account of customer demand.

The ensuing examples of concrete embodiments of the invention are given by way of explanation.

Example 1 (see Fig. 4):

The example explains the production of a continuously cast ingot of a  $\gamma$ -TiAl-based alloy of a composition of Ti-46.5Al-4 (Cr,Nb,Ta,B) (indicated in atomic percent) with a diameter of 180 mm and a length of 2,600 mm.

The first step consists in the production of four once-VAR-melted electrodes of a diameter of 150 mm and a length of 1,000 mm from pressed electrodes that contain all the alloying constituents in the form of Ti sponge, Al granules and suitable pre-alloys for Cr, Nb, Ta and B. The rods, which are not yet homogeneous, serve as electrodes for the manufacture of pre-homogenized, molten material by inductive melting in an HF coil. The electrodes are conical at the foot, the set angle being approximately 45°.

Upon inductive melting, an electrode is supplied from the depot that holds all the four electrodes to the HF melting coil of likewise conical design, and inductively melted. The melt originates on the entire surface of the cone, at the tip of the cone collecting in a melt stream in which the material is pre-homogenized. By the force of gravity, the melt arrives in the cold wall induction crucible which is located below the melting coil. The frequency at the outer oscillating circuit of the melting coil is 80.6 kHz. Uniform pre-heating of the dropping electrode by inductive heating (mean frequency approximately 500 Hz to 1 kHz) by way of an auxiliary coil, which is mounted above the melting coil, to temperatures below the melting point of the alloy (approximately 1300°C) helps obtain increased melting capacity of more than 50 kg/h. The electrode is rotated at a speed of 4 rpm, the withdrawal speed is approximately 12 mm/min.

15 The pre-homogenized molten material drops into a cold wall induction crucible with a bottom that can be drawn off downwards. The diameter of the crucible is 180 mm. The melt solidifies in the bottom area of the crucible and is continuously withdrawn downwards. Cooling of the melt upon ingot withdrawal takes place by means of water-cooled copper segments. The withdrawal rate amounts to approximately 1 mm/min. The average dwell time of the melt for homogenization in the cold wall induction crucible is approximately 20 min, which corresponds to a bath height of approximately 160 mm. The bath temperature is in the range of 1580°C and the frequency of the induction coil that surrounds the crucible amounts to 12 kHz.

Once the first electrode has melted off, the second electrode is moved into the required position and heated for melting, billet withdrawal being inter-

rupted for this period. Then the process is continued as specified until all the four electrodes of the depot have melted off.

Vacuum as well as protective gas execution of the method is conceivable.

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The ingot obtained has a diameter of approximately 180 mm and a total length of 2,600 mm and excels by excellent chemical and structural homogeneity. Local aluminum and titanium fluctuations are less than  $\pm 0.5$  atomic percent, those of the elements Cr, Nb and Ta being less than  $\pm 0.2$  atomic percent and the fluctuation of B being less than  $\pm 0.05$  atomic percent.

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Example 2 (see Fig. 5):

15 Example 2 differs from Example 1 by the kind and way of production of the molten material and supply to the KIT. The process is carried out under He protective gas. The PACHM process (plasma arc cold hearth melting) offers an alternative of inductive melting. In the present embodiment, the starting material in the form of once-VAR-melted electrodes corresponding to Example 1 is melted by an He plasma torch (150 kW) in a water-cooled copper crucible and led on via a water-cooled channel which is equally heated by an He plasma torch (150 kW). The amperage of the plasma torches above the cold hearth is approximately 500 A. The liquid alloying melt flows in the skull of its proper material towards an overflow above the  
20  
25 KIT, from where it flows continuously into the KIT. The starting material is continuously re-charged via a hydraulically triggered slope. The cold crucible has two principal functions: in addition to working as a reservoir

for pre-homogenized molten material, it serves as a place of deposit of undesired high-density and ceramic inclusions.

The process continues analogously to Example 1.

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The technical data given in the Examples do not restrict the scope of the invention in any way. In particular the number, type and capacity of the plasma torches, the cold crucible material, capacity and frequency ranges of the induction coils, diameters of the KIT, bath height of the melts in the

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KIT and feed and withdrawal rates can be varied within the scope of the prior art without any negative effect on the invention.

### Claims

1. A method of producing metallic and intermetallic alloy ingots by continuous or quasi-continuous billet withdrawal from a cold wall induction crucible, characterized in that the alloy material is supplied in a molten and pre-homogenized state continuously or quasi-continuously to a cold wall induction crucible.
2. A method according to claim 1, characterized in that inter-metallic  $\gamma$ -TiAl-based alloy ingots are produced.
3. A method according to claim 1 and 2, characterized in that the alloys are described by the following summation formula:  
$$\text{Ti}_x\text{Al}_y(\text{Cr,Mn,V})_u(\text{Zr,Cu,Nb,Ta,Mo,W,Ni})_v(\text{Si,B,C,Y})_w$$
with the concentrations of the alloying constituents being within the following ranges (in atomic percent):  
$$x = 100 - y - u - v - w$$
$$y = 40 \text{ to } 48, \text{ preferably } 44 \text{ to } 48$$
$$u = 0.5 \text{ to } 5$$
$$v = 0.1 \text{ to } 10 \text{ and}$$
$$w = 0.05 \text{ to } 1.$$
4. A method of producing metallic and intermetallic alloy ingots of high homogeneity and low porosity of any adjustable diameter according to claim 1, characterized in that it is based on the following sequence:
  - (i) producing electrodes by customarily mixing and compressing the selected starting materials;

- (ii) at least once remelting the electrodes obtained in step (i) in a conventional fusion-metallurgical process;
- (iii) inductively melting off the electrodes obtained in steps (i) and (ii) in a high frequency coil;
- 5 (iv) homogenizing the pre-homogenized, molten material obtained in step (iii) in a cold wall induction crucible; and
- (v) withdrawing the melt, solidified by cooling, from the cold wall induction crucible of step (iv) in the form of solidified ingots of freely adjustable diameters and lengths.

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5. A method according to claim 1, characterized in that it is based on the following sequence:

- (i) producing electrodes by conventionally mixing and compressing the selected starting materials;
- 15 (ii) at least once melting the electrodes obtained in step (i) by a conventional fusion-metallurgical method;
- (iii) producing a pre-homogenized, molten material of the electrode material obtained in step (ii) by melting off in a cold crucible plasma furnace;
- 20 (iv) homogenizing the pre-homogenized, molten material obtained in step (iii) in a cold wall induction crucible; and
- (v) withdrawing the melt, solidified by cooling, from the cold wall induction crucible of step (iv) in the form of cylindrical ingots of freely adjustable diameters and lengths.

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6. A method according to claims 1 to 4, characterized in that the melting process for producing the pre-homogenized, molten material takes place in a high frequency field of a frequency in the range of 70 to 300 kHz.



7. A method according to claims 1 to 4, characterized in that the temperature of the pre-homogenized, molten material ranges between 1400 to 1600°C.

5    8. A method according to claims 1 to 4, characterized in that the electrodes (iii) used for producing the molten, pre-homogenized material by means of an induction coil rotate preferably at a speed between 2 and 5 rpm.

9. A method according to claims 1 to 4, characterized in that the method is  
10    executed quasi-continuously by one or several electrodes, in case of inductive melting, being quasi-continuously fed while an ingot is simultaneously withdrawn from the cold wall induction crucible.

10. A method according to claims 1 to 4, characterized in that homogeniza-  
15    tion in the cold wall induction crucible in step (iv) takes place at a temperature of 1400 to 1700°C.

11. A method according to claims 1 to 4, characterized in that homogeniza-  
tion in the cold wall induction crucible in step (iv) takes place in a range of  
20    frequency of 4 to 20 kHz.

12. A method according to claims 1 to 4, characterized in that cooling the  
melt upon ingot withdrawal in step (v) takes place by the aid of water-  
cooled copper segments.

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13. A method according to claims 1 to 4, characterized in that the diameter of the ingots withdrawn in step (v) is in the range of 40 to 350 mm.

14.  $\gamma$ -TiAl-based alloy ingots produced according to claims 1 to [[1]]3,  
characterized by

- (a) a length to diameter ratio of  $> 12$ ;
- (b) homogeneity related to local macroscopic fluctuations of the aluminum  
5 and titanium of maximally  $\pm 0.5$  atomic percent; further metallic alloy-  
ing constituents of maximally  $\pm 0.2$  atomic percent; non-metallic alloy-  
ing additions (boron, carbon, silicon) of maximally  $\pm 0.05$  atomic per-  
cent.

Abstract

The invention relates to a method of producing metallic and intermetallic alloy ingots by continuous or quasi-continuous billet withdrawal from a  
5 cold wall crucible, which is characterized in that the alloy material is continuously or quasi-continuously supplied in a molten and pre-homogenized state to a cold wall induction crucible.